



# Effect of isochronal hydrogen annealing on surface roughness and threading dislocation density of epitaxial Ge films grown on Si

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## ABSTRACT

We report the effect of hydrogen annealing on the surface roughness and threading dislocation density (TDD) of germanium (Ge) films grown on silicon (Si) substrates by reduced-pressure chemical vapor deposition (RPCVD). The surface roughness initially decreased with an increase in the annealing temperature. At annealing temperatures greater than 650 °C the film thickness varied owing to surface undulations, leading to an increase in the surface roughness. Although high-temperature annealing at 850 °C is effective for reducing TDD, the surface roughness of a 150-nm-thick Ge film annealed at 650 °C reaches a minimum value (~0.7 nm).

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## 1. Introduction

Germanium (Ge)/silicon (Si) heterostructures are being increasingly used for the fabrication of novel devices that are compatible with Si-based technologies. In particular, Ge is a very promising material as a future channel material for nanoscale metal-oxide-semiconductor field-effect transistors (MOSFETs) due to its high mobility [1], and it finds an important application in the fabrication of photodetectors on Si [2]. Moreover, epitaxial growth of Ge on a Si substrate is an important method for producing suitable substrates for III–V-based devices [3]. For successful fabrication of these high-speed MOSFETs and optical devices, it is necessary to reduce the surface roughness and threading dislocation density (TDD) of Ge films.

Nayfeh et al. demonstrated that hydrogen annealing results in a dramatic improvement in the surface roughness of Ge films [4]. They found that annealing at 825 °C resulted in almost a 90% decrease in the surface roughness, i.e., from 25 nm to 2.9 nm. Further, they developed a novel technique that involved multiple growth and hydrogen annealing steps for depositing high-quality heteroepitaxial Ge films on Si [1]. Several research groups have used a two-step deposition method to grow thick Ge films with a smooth surface on Si [5–9]. In the first step, a 30–50-nm-thick seed layer is deposited in the layer-by-layer growth region at 330–400 °C. In the second step, the growth temperature is increased from 600 to 850 °C for the formation of a high-quality flat Ge film at a high deposition rate. Recently, Choi et al. grew high-quality, pure thick Ge films on Si substrates by using a combination of Nayfeh et al.'s method and the abovementioned two-

step deposition method [10]. They found that minimum values of the TDD and the root-mean-square (RMS) surface roughness ( $R_{\text{RMS}}$ ) of 1–2- $\mu\text{m}$ -thick samples are  $(0.8\text{--}1.0) \times 10^7 \text{ cm}^{-2}$  and 0.4–0.6 nm, respectively. Hydrogen annealing is thought to be the most important process not only in the multiple deposition and annealing steps but also during the second high-temperature growth step in the two-step deposition method; however, the net effect of this annealing on the resultant film remains to be clarified.

In this study, we deposited one 150-nm-thick Ge film on Si substrate by reduced-pressure chemical vapor deposition (RPCVD) and subsequently annealed in a hydrogen ambient. Since the two-step deposition method was not adopted for this purpose, the substrate was maintained at a low temperature (310–350 °C) during deposition. Therefore, this paper only reports the effect of isochronal hydrogen annealing on the reduction in the surface roughness and TDD of the deposited Ge films.

## 2. Experimental

The samples were grown and annealed in an industrial RPCVD system (Applied Materials Epi Centura). Si(100) substrates were first cleaned using a 4:1  $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$  mixture, and then with a 1:1:4 mixture of HCl,  $\text{H}_2\text{O}_2$ , and high-purity deionized (DI) water; subsequently, the substrates were rinsed with high-purity DI water. The substrates were then dipped into 2% HF for native oxide removal, rinsed with DI water, and immediately loaded into the reactor. After hydrogen annealing at 1100 °C, an epitaxial Si buffer layer was deposited on the Si substrate to reduce oxygen and carbon pile-up at the Ge/Si heterointerface [11]. Epitaxial Ge films (thickness: ~150 nm) were grown at 310 and 350 °C using a  $\text{GeH}_4\text{--H}_2$  gas mixture. Immediately after the epitaxial growth,

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hydrogen annealing was performed for 15 min in the temperature range of 450–850 °C at a pressure of 11 kPa. The surface roughness of the Ge films was determined by tapping-mode atomic force microscopy (AFM). The TDD of the films was determined by plan-view transmission electron microscopy (TEM). The dislocations formed in the films were analyzed by cross-sectional TEM observations.

### 3. Results and discussion

The growth mode of Ge on Si during RPCVD was found to be critically dependent on the substrate temperature ( $T_S$ ). Above 400 °C, large Ge islands were formed on the Si substrate [8,12]. The Ge layers grown below 400 °C were characterized by three different surface morphologies, as shown previously by Olubuyide et al. [9]. At low  $P_{\text{GeH}_4}$  partial pressures ( $P_{\text{GeH}_4}$ ), i.e.  $P_{\text{GeH}_4} < 2$  Pa, the growth of small islands with diameters of a few tens of nanometers occurred due to the suppression of adsorption and decomposition of  $\text{GeH}_4$  on Si–H bonds [13]. At  $P_{\text{GeH}_4} > 30$  Pa, the growth of large islands with diameters of a few hundred nanometers was favored. Between the two abovementioned pressure regimes, i.e., at  $P_{\text{GeH}_4} \sim 10$  Pa, a layer-by-layer growth region was found to exist.

Fig. 1(a) shows the  $5 \times 5 \mu\text{m}^2$  AFM scan of an as-deposited Ge film (sample A;  $T_S = 310$  °C;  $P_{\text{GeH}_4} = 32$  Pa). Fig. 1(b–f) show the AFM images of the Ge films after 15 min of isochronal hydrogen annealing in the temperature range of 450–850 °C. The growth of islands in the  $\langle 110 \rangle$  direction can be clearly observed in Fig. 1(a). Islands are formed on the flat film surface and  $R_{\text{RMS}}$  is 18.9 nm. After isochronal annealing at 450 °C, surface pits are formed along with small islands. Above 650 °C, only surface pits with a diameter of  $\sim 1 \mu\text{m}$  exist. Similar pits have often been observed on the surface of Ge films deposited by the previously mentioned two-step deposition method [14].

Fig. 2(a) shows the  $5 \times 5 \mu\text{m}^2$  AFM scan of an as-deposited Ge film (sample B;  $T_S = 350$  °C;  $P_{\text{GeH}_4} = 12$  Pa), while Fig. 2(b–f) show the AFM images of the Ge films after isochronal hydrogen annealing. Since the Ge film is deposited in the layer-by-layer growth region, its surface is very smooth ( $R_{\text{RMS}} = 1.2$  nm), and the film has a thickness of 150 nm (Fig. 2(a)). A small undulation with a wavelength of  $\sim 0.1 \mu\text{m}$  can be seen on the as-deposited Ge surface, and the surface of this film becomes smoother with an increase in the annealing temperature. After annealing at 450 and 550 °C, surface pits with a diameter

of  $\sim 0.5 \mu\text{m}$  appear on this film and the surface pit density of this sample was less than that of sample A. The surface undulations reappear at temperatures above 650 °C. The undulation wavelength increases with the annealing temperature.

Fig. 3(a) shows the cross-sectional TEM images of sample A before and after isochronal hydrogen annealing. Ge islands are formed on the as-deposited film surface, as seen in Fig. 1(a), with V-shaped defects in every island. Furthermore, threading dislocations and stacking faults, as well as misfit dislocations at the Ge/Si heterointerface, can be seen in the film. After annealing at 450 °C, the Ge islands are transformed into pits. Above 650 °C, the stacking faults and V-shaped defects disappear, while the misfit dislocations are mostly confined to the Ge/Si heterointerface and do not thread to the surface. Fig. 3(b) shows the cross-sectional TEM images of sample B before and after hydrogen annealing. No V-shaped defects are seen in the as-deposited film, and the film surface is smooth with no island formation. Above 650 °C, stacking faults are no longer observed, and the misfit dislocations are mostly confined to the Ge/Si heterointerface. In particular, at 850 °C, misfit dislocations are confined to the Ge/Si heterointerface or run parallel to it. A majority of the observed dislocations in the film annealed at 850 °C are 90° full-edge dislocations and the average spacing between the dislocations in Fig. 3(c) and (d) is  $\sim 10$  nm which is consisted with that reported previously [6,15].

Fig. 4 illustrate the dependence of  $R_{\text{RMS}}$  and the Z range (i.e.  $Z_{\text{max}} - Z_{\text{min}}$ ) on the annealing temperature, as determined by  $10 \times 10 \mu\text{m}^2$  AFM scans. The surface roughness of sample A initially decreased with an increase in the annealing temperature, reaching a minimum value at 650–750 °C and increased thereafter. However, Nayfeh et al. [4] had previously reported that  $R_{\text{RMS}}$  decreases monotonically with the annealing temperature (Fig. 4(a)). After annealing,  $R_{\text{RMS}}$  of sample B decreased, reaching a minimum of  $\sim 0.7$  nm at 650 °C. On the other hand, as the annealing temperature increased, the Z range of sample B initially increased because of pit formation and then decreased to a minimum value at 650 °C because of pit deformation (Fig. 2). Above 650 °C, the undulation wavelength increased, causing a corresponding increase in the Z range; this in turn led to an increase in  $R_{\text{RMS}}$ . In the case of sample A, the Z range increased with the annealing temperature because of pit formation, reaching a maximum between 550 and 650 °C, and then decreased monotonically. The reason of

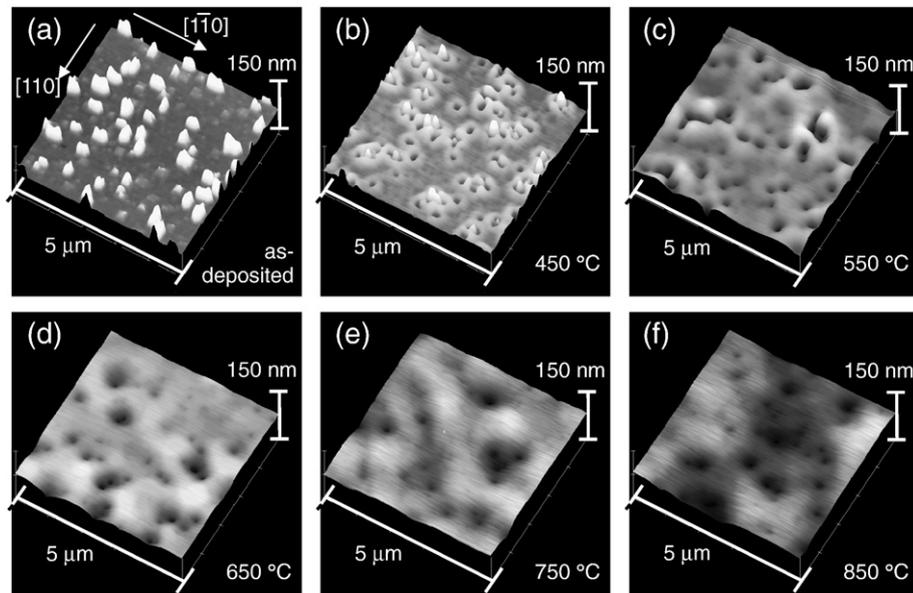
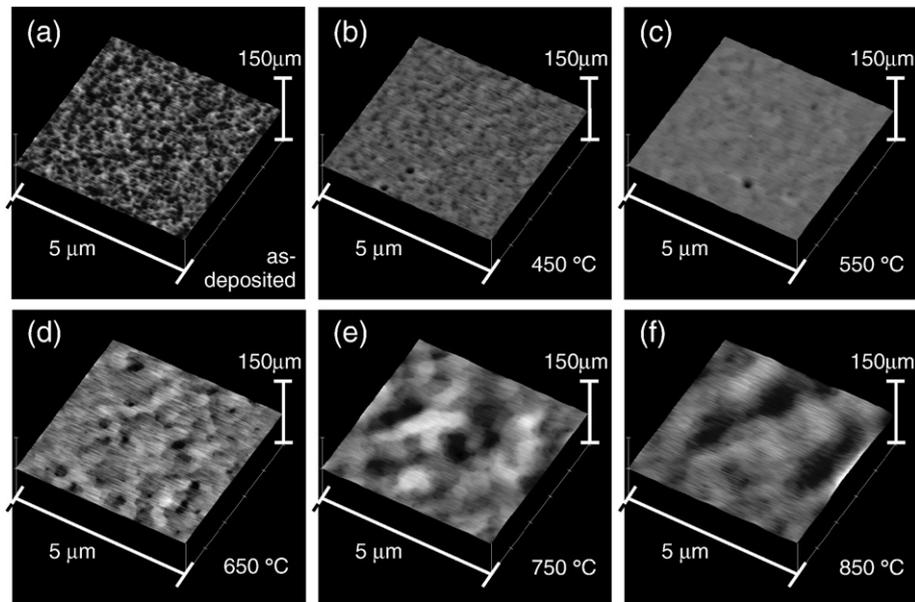


Fig. 1. AFM scans: (a) an as-deposited Ge film (sample A;  $T_S = 310$  °C;  $P_{\text{GeH}_4} = 32$  Pa); (b–f) Ge films after 15 min of isochronal hydrogen annealing at 450–850 °C.

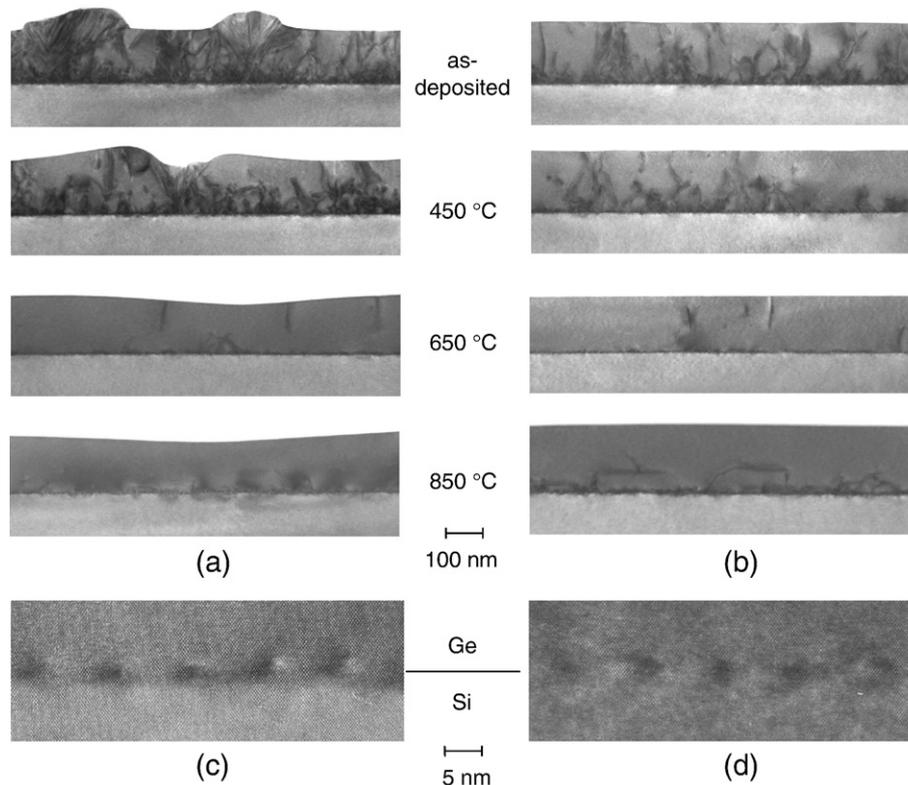


**Fig. 2.** AFM scans: (a) an as-deposited Ge film (sample B;  $T_S = 350$  °C;  $P_{\text{GeH}_4} = 12$  Pa); (b–f) Ge films after 15 min of isochronal hydrogen annealing at 450–850 °C.

this decrease at high annealing temperatures was more likely to be pit deformation rather than formation of long-wavelength (in  $\mu\text{m}$ ) surface undulations. However, the re-increase in  $R_{\text{RMS}}$  after high-temperature hydrogen annealing (Fig. 4(a)) indicated the ineffectiveness of high-temperature annealing in reducing the surface roughness.

The TDDs of the samples were determined by plan-view TEM (Fig. 5). Before annealing,  $R_{\text{RMS}}$  of sample B was approximately one order of magnitude smaller than that of sample A (Fig. 4); however, the TDDs of both these samples were approximately the same. Thus,

we concluded that island formation on the as-deposited film surface did not strongly affect the TDD. The TDDs of the samples reduced monotonically with an increase in the annealing temperature owing to coalescence and the glide of dislocations towards the edge of the substrate. The surface pit density measured by AFM is also shown in Fig. 5. Because of the presence of surface undulations, the surface pits could not be clearly observed in sample B at temperatures above 650 °C. Although the surface pit density of sample A also decreased monotonically with an increase in the annealing temperature, it was one or two orders of magnitude smaller than the TDD.



**Fig. 3.** Cross-sectional TEM images of as-deposited film and those of the films annealed at 450, 650, and 850 °C. (a): sample A; (b) sample B; (c) Ge/Si heterointerface of sample A annealed at 850 °C; (d) Ge/Si heterointerface of sample B annealed at 850 °C.

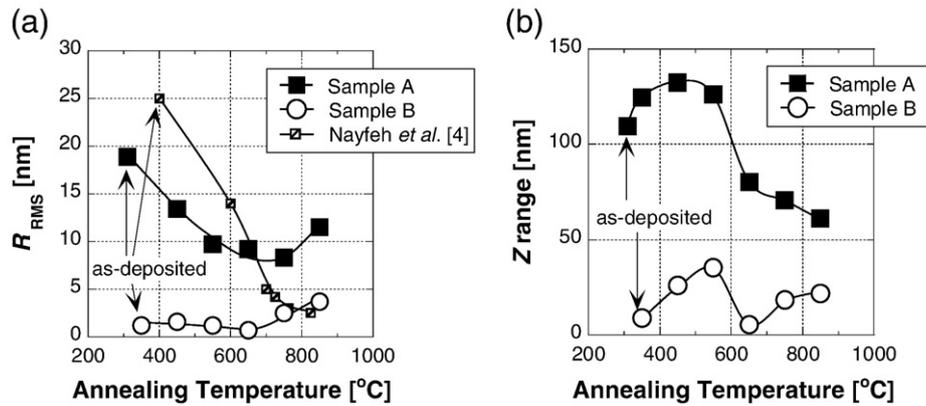


Fig. 4. (a): Dependence of  $R_{RMS}$  on annealing temperature. Sample A (■) is deposited at  $T_S=310^\circ\text{C}$  and  $P_{\text{GeH}_4}=32\text{ Pa}$  and sample B (○) is deposited at  $T_S=350^\circ\text{C}$  and  $P_{\text{GeH}_4}=12\text{ Pa}$ . Data reported by Nayfeh et al. (□) are also plotted. (b): Dependence of Z range on annealing temperature.

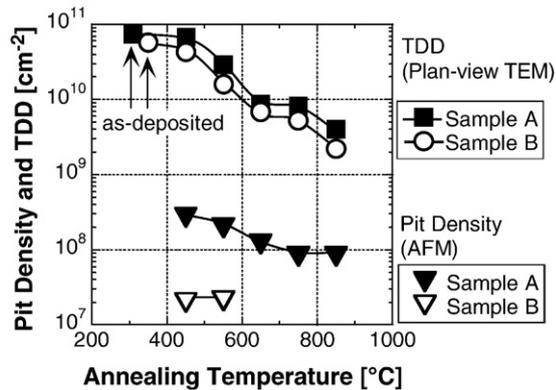


Fig. 5. Dependence of surface pit density (▼: sample A; ▽: sample B) and TDD (■: sample A; ○: sample B) of the Ge film on annealing temperature.

#### 4. Conclusion

Ge films were deposited on Si substrates by RPCVD and then annealed in a hydrogen ambient. After annealing, surface pits with a diameter of  $\sim \mu\text{m}$  appeared. The surface roughness initially decreased with an increase in the annealing temperature. At temperatures above  $650^\circ\text{C}$  the surface undulations appeared and undulation wavelength increased with the annealing temperature, causing variations in film thickness; this in turn led to an increase in the surface roughness. Although high-temperature annealing was effective in reducing the TDD, the surface roughness of the film annealed at  $650^\circ\text{C}$  reached a minimum value. Thus, we conclude that the optimum temperatures at which the surface roughness and TDD of the film reach a minimum value are different.

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